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Diacetyl and Acetylmethylcarbinol Production in the Manufacture of Unsalted Butter

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SUMMARY

During the ripening of sweet or neutralized sour cream in laboratory and semi-commercial trials there were some irregularities in the effects of various factors on the diacetyl and acetylmethylcarbinol contents of the cream, but generally the contents were increased by an increase in the acidity to which the cream was ripened, by addition of small amounts of citric acid to the cream and by agitation (shaking in the laboratory trials and revolving the coils in the semi-commercial trials) during the ripening. In some trials the contents were greatly influenced by the use of certain butter cultures, while in other trials they were not.

In general, as the diacetyl contents increased in the ripening cream the acetylmethylcarbinol contents also increased, but there were variations from this relationship. The occasional decreases in diacetyl contents often were accompanied by increases in acetylmethylcarbinol contents.

Some of the ripening procedures used with the cream were beneficial from the standpoint of score of the butter under certain holding conditions. These procedures included development of higher acidities in the cream, addition of citric acid to the cream and agitation of the cream during ripening.

With the semi-commercial churnings the ratios of the diacetyl contents of the cream to those of the corresponding butter were: minimum 1:0.142; maximum 1:0.709; and average 1:0.352. With acetylmethylcarbinol the ratios were 1:0.059; 1:0.683; and 1:0.217, respectively.

The diacetyl or acetylmethylcarbinol contents of the lots of butter in a series sometimes did not follow the same order as the contents of the lots of cream from which the butter was churned; the irregularities were of various types.

With the holding of butter 1 week at 36° to 40°F., 3 days at 60°F. plus 4 days at 36° to 40°F. or 1 month at 36° to 40°F., both increases and decreases in the diacetyl and acetylmethylcarbinol contents occurred. With each compound under each set of conditions the increases were more numerous than the decreases. After holding 3 days at 60°F. plus 4 days at 36° to 40°F., the diacetyl contents were higher in 75 percent of the comparisons, and the acetylmethylcarbinol contents were higher in 73 percent of the comparisons than in corresponding butter held 1 week at 36° to 40°F.

Diacetyl and Acetylmethylcarbinol Production in the Manufacture of Unsalted Butter[1]

By T. I. Hedrick and B. W. Hammer

High-flavored unsalted butter has increased greatly in commercial importance during recent years. This type of butter is churned for table use from either sweet or neutralized sour cream that has been ripened to a relatively high acidity after pasteurization and addition of butter culture. It is distinct from the unsalted butter used in ice cream mixes and certain other products; such butter is made without butter culture from cream having a low acidity at churning.

Without added salt it is necessary that the butter have a high flavor if it is to satisfy consumers. Accordingly, the problems with unsalted table butter differ from those with salted butter. Absence of salt permits development of acidities during cream ripening and holding of the butter which would be very objectionable with salted butter because of the probability of serious chemical defects appearing. Development of acidity in the cream favors production of flavor and also limits various types of bacterial deterioration.

Since diacetyl is such an important contributant to the desirable flavor and aroma of butter, the amount of diacetyl can be used as an index of flavor development in cream or butter. The common assumption that diacetyl is formed by the biological oxidation of acetylmethylcarbinol suggests the determination of acetylmethylcarbinol also. While there may be other compounds, for example volatile acids and esters of fatty acids, contributing to butter flavor, information with reference to them has not advanced to the point where analyses seem advisable.

The data reported herein deal (a) with effects of acidity, added citric acid, agitation, ripening temperature and different butter cultures on diacetyl and acetylmethylcarbinol production in cream ripened under laboratory conditions and also under semi-commercial conditions; and (b), in the case of butter

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[1]Project 127 of the Iowa Agricultural Experiment Station. The studies were financed in part through a grant from the Iowa State Brand Creameries Inc., of Mason City, Iowa.

made under semi-commercial conditions, with effects of various factors on flavor, with ratios of diacetyl and acetylmethylcarbinol contents of ripened cream and the corresponding butter and with changes in diacetyl and acetylmethylcarbinol contents of butter during holding under different conditions.

HISTORICAL

Studies of various investigators have established the importance of diacetyl as a flavor constituent of butter and have indicated various factors that influence its production in butter cultures by the normal butter culture organisms. The literature along these lines has been repeatedly reviewed and is not considered here.

The manufacture of butter without salt results in a product that from the standpoint of deterioration is definitely different from the usual salted butter. In unsalted butter objectionable chemical changes are less likely to occur than in salted butter; for example, fishiness is unusual in unsalted butter (5), and when it develops excessive amounts of copper often are involved. Without the restraining effect of salt, unsalted butter is relatively susceptible to growth of various organisms, including certain types that have a deteriorating action; however, with organisms that are not relatively acid tolerant, the acid present in the fresh butter, or produced in it during holding, may have a distinct inhibitory influence.

Minster (7) considered the presence of a few lactic acid organisms in unsalted butter as beneficial, since they tended to keep down growth of more objectionable species.

In unsalted butter held at 69.8°F., Nelson and Hammer (9) found large increases in the numbers of butter culture organisms with both plate and microscopic procedures. Striking evidence of the growth was the appearance of long chains of streptococci in the butter. Organisms other than streptococci also showed extensive growth. In unsalted butter held at —4°F., there were conspicuous decreases in the microscopic counts, the individual samples showing decreases from 7 to 98 percent (average 74%); the largest decreases occurred with samples having the highest counts when they were placed at —4°F. and the smallest decreases with samples having the lowest initial counts.

Slatter and Hammer (14) noted that in unsalted butter made from sweet cream with butter culture there commonly was a striking production of acetylmethylcarbinol plus diacetyl when the butter was held at temperatures permitting activity of the butter culture organisms; also, the pH of the butter markedly decreased. Production of the carbinol plus diacetyl varied widely with different lots of butter under the same conditions. The largest production occurred at the higher holding temperatures and in samples which developed the lowest pH values. Production usually was greater with a large addition of butter culture to the cream than with a small addition, and in some cases it was increased conspicuously by adding citric-acid-fermenting streptococci along with the usual butter culture. Occasionally the carbinol plus diacetyl decreased during the first few days of holding; this decrease sometimes was followed by an increase. Also, a striking production of the carbinol plus diacetyl sometimes was followed by a decrease.

Wiley (16) reported that at 40° or 65°F. deterioration of unsalted butter through bacterial action was not delayed by the presence of acid in the butter. At 0° or 14°F. the greatest deterioration was observed in butter from cream ripened to pH 5 (about 0.4% acid); butter from cream acidified to the same point by addition of pure lactic acid did not deteriorate to the same extent. Fat of the butter from the ripened cream showed notable oxidation after cold storage, but that from the acidified cream did not. In a later study Wiley (17) found that acidity, butter culture organisms, salt and low pasteurization temperatures each favored oxidation of the fat of butter during cold storage, while diacetyl and acetylmethylcarbinol did not. He concluded that the results indicate the presence in ripened pasteurized cream and in unripened raw cream of a fat-oxidizing enzyme which is most active at low pH values (about 5) and high salt concentrations.

In studies on changes in diacetyl and acetylmethylcarbinol contents of butter at various temperatures, Prill and Hammer (13) found that with salted butter which was frozen or at 36° to 45°F. only slight changes occurred; even at 70°F. the contents of the compounds were largely retained for considerable periods. With unsalted butter at —10° to 0°F. the changes were relatively slight, but at 36° to 45°F. or at 70°F. significant changes occurred, both increases and decreases being involved.

GENERAL PROCEDURE

Trials were carried out both in the bacteriological laboratory and in the butter manufacturing plant of the Iowa State College, although even in the latter case the trials were on a relatively small scale (semi-commercial). The general methods employed in the two types of trials are as follows:

LABORATORY TRIALS

The cream was pasteurized in the regular operations of the butter plant, a 300-gallon vat and 150°F. for 30 minutes being employed. The butterfat contents ranged from 32 to 37 percent. The sweet cream had an initial acidity of 0.20 percent or less; after pasteurization it was neutralized (at about 120°F.) to approximately 0.10 percent acid with sodium sesqui-carbonate. The sour cream had an initial acidity of 0.40 to 0.50 percent; before pasteurization this was reduced (at about 90°F.) to 0.25 percent with "Alkali Special" and after pasteurization the reduction was continued (at about 120°F.) to 0.15 percent with sodium sesqui-carbonate. The cream was cooled to 36°F., and a portion was removed to a container that had been thoroughly steamed. Five percent butter culture by weight was added, and then 800-gram portions of the mixture were put into sterile quart jars having glass tops. The portions were warmed to the desired ripening temperatures and incubated at such temperatures until the desired acidities had developed. In order to avoid agitation of the 800-gram portions during the ripening, which would influence the diacetyl contents by incorporating air, a 250-gram portion of the mixture, in a pint bottle, was held at each of the ripening temperatures and used to follow the increase in acid. When the desired acidity was reached in a 250-gram portion, 300 grams were removed from the 800-gram portion and the various analyses made. The remaining 500 grams were churned in a small laboratory churn, but the difficulty of controlling the operation resulted in such irregularities in the retention of buttermilk in the butter that the data are not considered in detail.

SEMI-COMMERCIAL TRIALS

The cream was pasteurized in the regular operations of the butter laboratory, either in a 300-gallon vat at 150°F. for 30 minutes or with a vacreator (2) using a temperature of 198°F. and a final vacuum of about 28 inches. Fat contents, acidities and neutralization procedures were essentially the same as with the cream employed in the laboratory trials. The pasteurized cream was cooled to 36°F.

For each churning 150 pounds of cream was transferred to a small vat, and 5 percent butter culture by weight was added. Usually the mixture of cream and culture was heated to the ripening temperature and held over night, but in trials on effect of temperature the ripening period varied. The cream increased in temperature (sometimes as much as 10°F.) during the ripening. When the ripening was complete the temperature was lowered for churning to approximately 48°F., and a sample was taken for the various analyses.

Each lot of cream was churned in a small single roll churn having a normal capacity of about 60 pounds of butter. The cream was churned until the butter granules were about the size of peas. After draining the buttermilk, the granules were rinsed with water until the water from the drain did not have a milky appearance. Two washings with water about 5°F. colder than the buttermilk were employed. The free moisture was drained, and an attempt was made to incorporate approximately 17 percent moisture in the finished butter.

Several 1-pound samples of butter were taken from each churning and wrapped in parchment paper. These were held under different conditions for various periods and then examined.

ANALYTICAL METHODS

ACID AND pH DETERMINATIONS

Acid determinations were made by titrating 9 grams of cream without dilution, using 0.1 normal sodium hydroxide and phenolphthalein; results were calculated as lactic acid to the nearest 0.01 percent. pH determinations were made with a potentiometer, a modified quinhydrone electrode and a calomel half-cell; values were calculated to the nearest 0.01 of a pH unit.

DIACETYL AND ACETYLMETHYLCARBINOL DETERMINATIONS

Cream and butter were analyzed for diacetyl and acetylmethylcarbinol by the colorimetric method of Prill and Hammer (10), with a few minor modifications used by Hoecker and Hammer (4). Also, the color intensities were determined with a Klett-Summerson photoelectric colorimeter. Results were recorded as parts per million (p.p.m.) of diacetyl or acetylmethylcarbinol. Actually the p.p.m. of acetylmethylcarbinol represent acetylmethylcarbinol plus diacetyl, but since the amount of diacetyl is insignificant compared to the amount of carbinol, this procedure did not materially affect the acetylmethylcarbinol values.

EXPERIMENTAL

EFFECTS OF VARIOUS FACTORS ON DIACETYL AND ACETYLMETHYLCARBINOL CONTENTS OF RIPENED CREAM

EFFECT OF RIPENING CREAM TO VARIOUS ACIDITIES

LABORATORY TRIALS

Results of 36 trials on sweet cream ripened to various acidities at temperatures from 50° to 70°F. are given in table 1. Each trial included either three or four acidities; in six trials a mixture of cream and culture also was held at 32°F. for a normal ripening period.

As the acidity of the cream increased, the diacetyl content increased in 29 trials (81%), and the acetylmethylcarbinol content increased in 32 trials (89%). Most of the increases were significant, but some of them were very small. Both diacetyl and acetylmethylcarbinol increases were small in the pH range of 6.5 to 5.5.

The diacetyl content decreased in two trials (6%). One of the decreases was small, from 0.57 to 0.44 p.p.m.; the other was larger, from 0.46 to 0.19 p.p.m., and may have been caused by an abnormal culture. The acetylmethylcarbinol content also decreased in two trials (6%), the decrease being large in each case; one of the decreases was in a trial that showed a decrease in diacetyl content.

An increase in diacetyl content followed by a decrease occurred in three trials (8%). One of the decreases was small, being 0.15 p.p.m.; and one was rather large, being 0.46 p.p.m. In the three trials corresponding changes in acetylmethylcarbinol did not occur, but in another trial (3%) the carbinol content increased and then decreased very slightly, from 29.8 to 29.0 p.p.m.

There was an increase in diacetyl content, then a decrease and finally an increase in two trials (6%) and a similar series of changes in carbinol in one trial (3%). The decreases were small in all cases, and in one of the changes in diacetyl the final increase was large.

The diacetyl contents of the six lots of cream plus butter culture held during a normal ripening period at 32°F. ranged from 0.12 to 0.28 p.p.m. with an average of 0.22 p.p.m.; the acetylmethylcarbinol contents varied from 3.5 to 16.5 p.p.m. and averaged 10.6 p.p.m.

Table 2 gives results of three trials on neutralized sour cream ripened to various acidities at temperatures from 58° to 62°F. Three acidities were used in each of two trials, and four acidities were employed in one trial; in each trial a mixture of cream and culture was held at 32°F. for the normal ripening period.

An increase in acidity resulted in an increase in diacetyl and also in acetylmethylcarbinol in each case. The increases in diacetyl regularly were conspicuous, and in two of the trials the increases in acetylmethylcarbinol were large.

The diacetyl and acetylmethycarbinol contents of the three lots of neutralized sour cream plus butter culture held at 32°F. generally were higher than those of sweet cream plus culture, the average diacetyl content being 0.42 p.p.m. (range from 0.35 to 0.55 p.p.m.) compared to 0.22 p.p.m. for the sweet cream and the average carbinol content being 22.1 p.p.m. (range from 20.9 to 23.3 p.p.m.) compared to 10.6 p.p.m. for the sweet cream.

This relationship probably is explained by the development of the two compounds during the souring of the cream.

SEMI-COMMERCIAL TRIALS

Results of 10 trials on cream (seven on sweet cream, three on sour neutralized cream) ripened to various acidities at temperatures from 50° to 55°F. are presented in table 3. Three acidities were employed in three of the trials with sweet cream and two acidities were used in the remaining trials. In two trials with sour cream, determinations were made on the cream just after pasteurization and again after the addition of culture.

With an increase in acidity there was an increase in diacetyl in nine trials (90%) and in acetylmethylcarbinol in seven trials (70%). Some of the increases were small. The diacetyl decreased in one trial (10%), the drop being from 1.88 to 1.49 p.p.m. with a change in acidity from 0.37 to 0.49 percent. The acetylmethylcarbinol decreased in three trials (30%), in one instance after a striking increase. Two of these trials involved sour cream in which the citric acid may have been completely fermented; the cream had relatively high diacetyl and acetylmethylcarbinol contents before the addition of butter culture, containing 0.21 p.p.m. diacetyl and 21.7 p.p.m. acetylmethylcarbinol in one trial and 0.56 and 29.8 p.p.m., respectively, in the other. With no citric acid present reduction of diacetyl and acetylmethylcarbinol by the citric-acid-fermenting organisms in butter cultures would be expected.

EFFECT OF ADDED CITRIC ACID

In the trials involving addition of citric acid to the cream, the acid was added as a sterile aqueous solution at the time of inoculating the butter culture.

LABORATORY TRIALS

Fourteen trials were carried out in which sweet cream was ripened with and without added citric acid to about the same acidity at temperatures from 54° to 60°F.; the amount of citric acid used was 0.05 percent except in one trial where it was 0.10 percent. Table 4 gives the results.

Addition of citric acid resulted in a higher diacetyl content in all the trials (100%) and in a higher acetylmethylcarbinol content in 12 trials (86%). With both diacetyl and acetylmethylcarbinol some of the increases were very small.

Eight trials were carried out in which neutralized sour cream was ripened with and without citric acid to about the same acidity at temperatures from 58° to 63°F.; in six of the trials three quantities of citric acid were used and in two trials only one quantity was employed. The results are given in table 5.

In six trials (75%) added citric acid resulted in an increase in the diacetyl content that, in the four trials involving three amounts of acid, was roughly in proportion to the amount added. In one trial (12.5%) 0.05 and 0.10 percent added citric acid did not increase the diacetyl content but 0.15 percent did, and in one trial (12.5%) 0.10 and 0.15 percent added acid gave the same diacetyl content. The added citric acid regularly increased the acetylmethylcarbinol content, and in the trials in which three amounts were used the increase was roughly in proportion to the amount added.

SEMI-COMMERCIAL TRIALS

Data on seven trials (four on sweet cream and three on neutralized sour cream) in which cream was ripened with and without added citric acid (0.05%) to about the same acidity at temperatures from 50° to 54°F. are given in table 6.

The added citric acid increased the diacetyl content in six comparisons (86%). The increases varied widely, with three of them being 0.20 p.p.m. or less. The one decrease in diacetyl content was very small, being 0.13 p.p.m. Added citric acid increased the acetylmethylcarbinol content in three comparisons (43%), the increases ranging from 12.8 to 34.8 p.p.m. In one trial (14%) the carbinol content was the same with and without added citric acid. In the three comparisons (43%) in which addition of the acid resulted in a decrease in the carbinol content, the decreases ranged from 4.0 to 15.8 p.p.m.

EFFECT OF AGITATION DURING RIPENING

LABORATORY TRIALS

In nine trials (seven with sweet cream and two with neutralized sour cream) cream was ripened with and without agitation at 60°, 63° or 70° F. Cream held at 60° or 63° F. was agitated three times during the ripening and that held at 70°F. was agitated every hour for 3 or 4 hours. Table 7 gives the data.

In each of the trials the diacetyl and acetylmethylcarbinol contents of the agitated cream were much higher than those of the unagitated cream. Essentially the same acidity developed with and without agitation.

The effect of agitation with added citric acid was studied in six trials (four with sweet cream and two with neutralized sour cream). One-tenth percent citric acid was used, and the ripening temperature was 60°, 63° or 70°F. The results are presented in table 8.

As without addition of citric acid, agitation greatly increased both the diacetyl and acetylmethylcarbinol contents. It had no appreciable effect on the acid development.

SEMI-COMMERCIAL TRIALS

Five trials were carried out (three with sweet cream and two with neutralized sour cream). The cream with the butter culture added was placed in the vats late in the afternoon and ripened over night. The next morning, approximately 3 hours before the ripening was complete, the cream was agitated by revolving the coils for one-half hour. Since the vats were only one-third full, the agitation was relatively vigorous. The data are given in table 9.

As in the laboratory trials the diacetyl and acetylmethylcarbinol contents were greatly increased by the agitation. The acid development was not appreciably influenced.

EFFECT OF RIPENING TEMPERATURE

LABORATORY TRIALS

In 18 trials sweet cream was ripened at 50°, 60° and 70°F. For each trial cream containing culture was warmed to these temperatures and ripened in constant temperature incubators to approximately the same acidity. The ripening periods varied from 5 to 45 hours, depending on the temperature and on the degree of acidity desired. Determinations of diacetyl and acetylmethylcarbinol were made soon after the desired acidity was reached. Table 10 presents the data.

The highest diacetyl content developed at 50°F. and the lowest diacetyl content at 70°F. in 14 (78%) of the trials;[2] the same relationship occurred with acetylmethylcarbinol in 11 trials (61%). Both diacetyl and acetylmethylcarbinol production were highest at 50°F. and lowest at 60°F. in two trials (11%). The highest diacetyl content was produced at 60°F. and the lowest at 70°F. in one trial (6%), and the same relationship occurred with the carbinol content in five trials (28%). Diacetyl production was highest at 70°F. and lowest at 60°F. in one trial (6%). Variations in acetylmethylcarbinol production at the three ripening temperatures were small and definitely less on a percentage basis than variations in diacetyl production.

SEMI-COMMERCIAL TRIALS

Since it was impossible to control the ripening temperature adequately in the small vats, cream containing culture was warmed to the desired temperature and then drawn into 10-gallon cans. The cans were held in a refrigerator at about the desired temperature until the desired acidity was reached. The temperature comparisons regularly involved cream ripened at 62° and 70°F. to about the same acidity; 55°F. was used only once because it required such an extended ripening period.

[2]Equal diacetyl and acetylmethylcarbinol values are not considered separately but are fitted into the general statements.

On reaching the desired acidity, cream ripened at 70°F. was cooled by placing the cans in ice water and stirring; after reaching 45°F. the cream was held over night at 36° to 40°F. The next morning the cream was warmed to 48°F. and churned. The cream ripened at 62°F. reached the desired acidity after ripening over night. The cans were placed in ice water, cooled to 48°F. and the cream then churned. Four trials were carried out, three with sweet cream and one with neutralized sour cream. Table 11 presents the data.

Diacetyl production in the cream ripened at 62°F. was larger than in cream ripened at 70°F. in two trials (50%) and smaller in two trials (50%). Acetylmethylcarbinol production showed similar variations. The differences in production of diacetyl and acetylmethylcarbinol in a comparison commonly were small, but in one trial involving a relatively low acidity the differences were large. Ripening at 55°F. gave slightly lower diacetyl and acetylmethylcarbinol contents in the one trial in which it was used.

In three trials a special vat of cream was ripened along with the cream used for the temperature comparisons; the treatment of the cream consisted of adding 0.1 percent citric acid and agitating during the ripening over night to an acidity of 0.45 percent. Diacetyl and acetylmethylcarbinol production in this cream was much larger in each trial than in the cream ripened at either 70°F. or 62°F.

EFFECT OF DIFFERENT BUTTER CULTURES

In various trials, both laboratory and semi-commercial, different butter cultures were employed to ripen cream, which sometimes was sweet and sometimes was sour. In certain trials the cultures came from plants receiving commercial cultures from the same laboratory, and in other trials they came from plants receiving commercial cultures from different laboratories. The cultures from the plants were transferred, usually twice, to have them in an active condition before they were inoculated into the cream. In each trial an attempt was made to have the different cultures develop essentially the same acidity in the cream, and the general conditions of ripening were kept uniform except that the incubation periods necessarily varied because of differences in the rates of acid development by the cultures.

The diacetyl and acetylmethylcarbinol contents developed by the different cultures varied rather widely in some trials and were rather uniform in others. When the same cultures were used in several trials they did not always rank the same on the basis of production of diacetyl or acetylmethylcarbinol, and the variations were not explainable on the basis of the slight differences in acidities of the ripened cream. Also, with the same cultures being used in several trials, all of them sometimes showed relatively high values in a trial and sometimes relatively low values.

RELATIONSHIP OF DIACETYL AND ACETYLMETHYLCARBINOL CONTENTS OF RIPENED CREAM

In general, as the diacetyl increased during the ripening of cream the acetylmethylcarbinol also increased, but in some instances an increase in diacetyl was accompanied by a decrease in the carbinol. Commonly, increases in diacetyl and acetylmethylcarbinol were in the same general proportion, but again there were exceptions to this relationship. The occasional decreases in diacetyl often were accompanied by increases in acetylmethylcarbinol, and when there was no change in diacetyl there often was an increase in the carbinol.

EFFECT OF VARIOUS RIPENING PROCEDURES ON FLAVOR OF UNSALTED BUTTER

Much of the unsalted butter made under semi-commercial conditions from cream ripened with various procedures was scored for flavor after 1 week at 36° to 40°F.; after 1 month at 36° to 40°F.; and after 6 months at —10° to 0°F. Usually two judges[3] scored the butter without knowledge of its identity, but in a few instances only one judge was available. Since the scoring of unsalted butter is not well standardized, comparative scores rather than absolute scores are given.

EFFECT OF RIPENING ACIDITY

AFTER 1 WEEK AT 36° TO 40°F.

Ripening the cream to the higher of the two acidites used in each trial gave butter which scored 0.25 to 0.75 of a point higher than that from the cream ripened to the lower acidity in five (71%) of the seven trials. There was no difference in score in one trial (14%) in which the acidities of the two lots of cream differed only 0.04 percent (0.31 and 0.35%); both lots of butter were criticized as tallowy and this off-flavor probably tended to mask the desirable flavor. With the higher acidity the butter scored 0.25 of a point lower in one trial (14%) in which the difference in acidities of the two lots of cream was small although both were relatively high (0.48 and 0.51%).

In the two trials in which the lots of cream had the greatest differences in acidity, 0.08 percent (0.38 and 0.46%) and 0.12 percent (0.37 and 0.49%), variations in the scores of the butter

[3]Dr. N. E. Fabricius and Dr. H. C. Olson did much of the scoring.

were the largest, being 0.75 of a point higher in each case with the higher cream acidity.

AFTER 1 MONTH AT 36° TO 40° F.

With the higher acidity the butter scored 0.25 to 0.5 of a point higher in three trials (43%). No difference in score was noted in two trials (29%). With the higher acidity the butter scored 0.5 of a point lower in two trials (29%). After the holding the scores were 0.25 to 1.5 points lower than after only 1 week at 36° to 40°F. except in one instance in which the score of the butter made from the higher acid cream increased 0.25 point.

AFTER 6 MONTHS AT —10° TO 0° F.

Ripening to the higher acidity gave butter which scored from 0.25 to 0.75 of a point higher in all the trials (100%). Following the holding the scores usually were 0.25 to 2.5 points lower than after 1 week at 36° to 40°F., but there were increases in score (0.5 to 1.0 point) in three instances, one involving butter from the lower acid cream and two involving butter from the higher acid cream.

EFFECT OF ADDED CITRIC ACID

AFTER 1 WEEK AT 36° TO 40° F.

Addition of citric acid to the cream before ripening resulted in a score increase of 0.25 to 0.5 of a point in four (57%) of the seven trials. No difference in score was noted in three trials (43%); in two of these sour cream was used and various off-flavors tended to mask the desirable flavor, while in the other addition of citric acid resulted in such a slight flavor improvement that no higher score was given.

AFTER 1 MONTH AT 36° TO 40° F.

With added citric acid there was a score improvement of 0.25 to 0.75 of a point in six trials (86%). There was no difference in score in one trial (14%). Following the holding the scores commonly were 0.25 to 2.0 points lower than after only 1 week at 36° to 40°F., but with three lots of butter, one made without acid added to the cream and two made with added acid, the scores increased from 0.25 to 0.75 of a point.

AFTER 6 MONTHS AT —10° TO 0° F.

Addition of citric acid resulted in a score increase of 0.5 to 0.75 of a point in six trials (86%). It resulted in a score decrease of 0.5 of a point in one trial (14%). After the holding the butter usually scored 0.5 to 2.25 points lower than after 1 week at 36° to 40°F., although in three instances, one involving butter made without acid added to the cream and two involving butter made with added acid, the scores increased from 0.5 to 1.25 points.

EFFECT OF AGITATION DURING RIPENING

AFTER 1 WEEK AT 36° TO 40° F.

Agitation of the cream during ripening gave a score increase of 0.25 to 0.75 of a point in three (60%) of the five trials. There was no difference in score in two trials (40%); this may have been due to off-flavors in the butter which tended to mask the desirable flavor.

AFTER 1 MONTH AT 36° TO 40° F.

With agitation of the cream there was a score increase of 0.5 to 0.75 of a point in two trials (40%). No difference in score occurred in two trials (40%); there seemed to be a tendency for some of the butter from agitated cream to have a slight oxidized flavor. Agitation resulted in a score decrease of 0.25 of a point in one trial (20%). After the holding the butter commonly scored 0.25 to 1.5 points lower than after only 1 week at 36° to 40° F., but in two instances, one involving butter from unagitated cream and one involving butter from agitated cream, there were score increases; these were 0.25 and 0.75 of a point.

AFTER 6 MONTHS AT —10° TO 0° F.

Agitation of the cream resulted in a score increase in four trials (80%); three of the increases were 1.0 point, and the other was 0.25 of a point. There was no difference in score in one trial (20%). Following the holding the scores were 0.25 to 2.0 points lower than after 1 week at 36° to 40° F.

EFFECT OF RIPENING TEMPERATURE

AFTER 1 WEEK AT 36° TO 40° F.

Ripening of the cream at 62° F., rather than at 70° F., gave a score increase of 0.25 to 0.75 of a point in two (50%) of the four trials. There was no difference in score in one trial (25%). Ripening at 62° F. resulted in a score decrease of 0.5 of a point in one trial (25%).

AFTER 1 MONTH AT 36° TO 40° F.

With ripening at 62° F. there was a score increase of 0.5 to 0.75 of a point in each of the three trials (100%). Following the holding the butter scored from 0.25 to 0.75 of a point lower than after only 1 week at 36° to 40° F. except in two cases involving butter made from cream ripened at 62° F.; in one of these cases there was no change in score while in the other there was a score increase of 0.25 of a point.

AFTER 6 MONTHS AT —10° TO 0°F.

Ripening at 62°F. gave a score increase of 0.5 of a point in each of two trials (50%). It gave a score decrease of 0.75 and 1.0 point in two trials (50%). After the holding the scores were 1.0 to 2.75 points lower than after 1 week at 36° to 40°F.

GENERAL RESULTS

The general effects of the various cream ripening procedures on the flavor of the unsalted butter are shown in the following summary:

Factor studied	Butter held		
	1 week 36° to 40°F.	1 month 36° to 40°F.	6 months —10° to 0°F.
	No. of trials showing changes		
Ripening acidity			
Higher acidity gave higher score	5	3	7
Higher acidity gave same score	1	2	0
Higher acidity gave lower score	1	2	0
Total	7	7	7
Addition of citric acid to cream			
Citric acid gave higher score	4	6	6
Citric acid gave same score	3	1	0
Citric acid gave lower score	0	0	1
Total	7	7	7
Agitation during ripening of cream			
Agitation gave higher score	3	2	4
Agitation gave same score	2	2	1
Agitation gave lower score	0	1	0
Total	5	5	5
Ripening temperature, 62° and 70°F.			
62°F. gave higher score	2	3	2
62°F. gave same score	1	0	0
62°F. gave lower score	1	0	2
Total	4	3	4

The data in the summary, together with the actual differences in scores of comparative lots of butter, were analyzed with the usual statistical methods, and the results used in the interpretations. In some of the comparisons a particular ripening procedure evidently was beneficial from the standpoint of the score of the butter under certain holding conditions, while in others it was not.

The higher acidity in the cream was especially useful with the butter held 6 months at —10° to 0°F. and was of some value with the butter held 1 week at 36° to 40°F.; it was of little value with the butter held 1 month at 36° to 40°F., presumably because during this holding there was an opportunity for extensive growth of the butter culture organisms. Addition of citric acid apparently had a desirable effect under all the holding conditions, as would be expected since this compound is a source of flavoring constituents of butter. Agitation had its most desirable effect with the butter held 6 months at —10° to 0°F. and its least desirable effect with the butter held 1 month at 36° to 40°F. Ripening at 62°F., rather than 70°F., evidently had no desirable effect with the butter held 6 months at —10° to 0°F. or 1 week at 36° to 40°F., but was definitely beneficial with the butter held 1 month at 36° to 40°F.

RATIOS OF DIACETYL AND ACETYLMETHYLCARBINOL CONTENTS OF THE RIPENED CREAM AND OF THE CORRESPONDING BUTTER

With 74 semi-commercial churnings (some not included in tables) the minimum, maximum and average ratios of the diacetyl and the acetylmethylcarbinol contents of the ripened cream and of the corresponding butter are as follows:

	Ratio of content of cream to content of butter		
	Minimum	Maximum	Average
Diacetyl	1:0.142	1:0.709	1:0.352
Acetylmethylcarbinol	1:0.059	1:0.683	1:0.217

The average ratio was considerably higher with diacetyl than with acetylmethylcarbinol; the less significant minimum and maximum ratios also were higher with diacetyl.

When the data are divided into groups on the basis of the factor whose effect on the diacetyl and acetylmethylcarbinol contents of ripening cream was being studied, the following values are obtained:

Factor studied	No. trials	Ratio of content of cream to content of butter		
		Minimum	Maximum	Average
Ripening acidity	23			
Diacetyl		1:0.164	1:0.652	1:0.390
Acetylmethylcarbinol		1:0.065	1:0.385	1:0.215
Added citric acid	14			
Diacetyl		1:0.142	1:0.547	1:0.328
Acetylmethylcarbinol		1:0.105	1:0.625	1:0.215
Agitation	10			
Diacetyl		1:0.165	1:0.395	1:0.300
Acetylmethylcarbinol		1:0.059	1:0.297	1:0.179
Ripening temperature	12			
Diacetyl		1:0.206	1:0.709	1:0.388
Acetylmethylcarbinol		1:0.107	1:0.683	1:0.244
Different butter cultures	15			
Diacetyl		1:0.210	1:0.486	1:0.322
Acetylmethylcarbinol		1:0.152	1:0.391	1:0.227

With each of the groups the average ratio was considerably higher with diacetyl than with acetylmethylcarbinol. There was reasonably close agreement between the values for the different groups; the lowest average ratio for both diacetyl and the carbinol occurred when the effect of agitation was investigated. The minimum and maximum ratios also were higher with diacetyl than with the carbinol except in the case of the maximum values when the effect of added citric acid was studied; also, there was little difference between the minimum values for diacetyl and acetylmethylcarbinol when the effect of added citric acid was investigated and little difference between the maximum values when the effect of ripening temperature was studied.

As already indicated the difficulty in controlling the churning of small volumes of cream in the laboratory resulted in such irregularities in the retention of buttermilk in the butter that the data are not considered in detail. This irregularity is suggested by the following ratios of the diacetyl and acetylmethylcarbinol contents of the cream and of the butter with 309 laboratory churnings (some not included in tables):

	Ratio of content of cream to content of butter		
	Minimum	Maximum	Average
Diacetyl	1:0.111	1:2.080	1:0.725
Acetylmethylcarbinol	1:0.054	1:0.961	1:0.339

The average ratios are considerably higher than with the 74 semi-commercial churnings; the maximum ratios also are higher while the minimum ratios are slightly lower. The values regularly were higher for diacetyl than for the carbinol.

CHANGES IN DIACETYL AND ACETYLMETHYLCARBINOL CONTENTS DURING HOLDING OF BUTTER

As would be expected from the variations in the ratios of diacetyl and acetylmethylcarbinol contents of ripened cream and of the corresponding butter, the diacetyl and acetylmethylcarbinol contents of a series of lots of butter sometimes did not follow the same order as the contents of the lots of cream from which the butter was churned. The irregularities were of all possible types. In some cases the diacetyl or acetylmethylcarbinol content of a lot of butter was lower than that of an earlier lot in the series although the content of the cream was higher. In other cases a value for diacetyl or acetylmethylcarbinol was lower than the values on the lots preceding and following it in the series, even when there was no drop in the value for the cream. Still other types of variations occurred. Because of these irregularities the diacetyl and acetylmethylcarbinol contents of the lots of butter are not given in detail. The diacetyl contents of the semi-commercial churnings of butter ranged from 0.16 to 1.46 p.p.m., while the acetylmethylcarbinol contents varied from 2.9 to 24.0 p.p.m. The highest values for both diacetyl and acetylmethylcarbinol were obtained on a lot of butter made from cream ripened with agitation after addition of 0.1 percent citric acid. Considerably higher values for diacetyl and acetylmethylcarbinol were obtained on butter from the laboratory churnings, but these undoubtedly were influenced by a high retention of buttermilk.

Changes in the diacetyl and acetylmethylcarbinol contents of butter during holding are of importance because of their probable relationship to development or disappearance of flavor. Changes in the contents of some of the semi-commercial churnings during 1 week at 36° to 40°F., during 3 days at 60°F. plus 4 days at 36° to 40°F. and during 1 month at 36° to 40°F. are shown in the following summary:

Compound and type of change	Holding conditions: 1 week 36° to 40° F.	3 days 60° F. plus 4 days 40° F.	1 month 36° to 40° F.
	No. of samples showing changes		
Diacetyl			
Increases total	47	45	39
0.10 p.p.m. or less	11	5	6
.11 to .20 p.p.m.	9	7	6
.21 to .30 p.p.m.	17	6	7
.31 to .92 p.p.m.	10	27	20
No change total	2	0	0
Decreases total	7	11	4
0.10 p.p.m. or less	2	6	1
.11 to .20 p.p.m.	2	1	2
.21 to .30 p.p.m.	0	0	1
.31 to .78 p.p.m.	3	4	0
Acetylmethylcarbinol			
Increases total	38	37	29
1.0 p.p.m. or less	7	3	4
1.1 to 3.0 p.p.m.	21	9	12
3.1 to 7.0 p.p.m.	9	11	11
7.0 to 23.0 p.p.m.	1	14	2
No change total	0	0	1
Decreases total	18	18	14
1.0 p.p.m. or less	5	4	2
1.1 to 3.0 p.p.m.	11	6	9
3.1 to 7.0 p.p.m.	2	4	3
7.1 to 11.5 p.p.m.	0	4	0

Under each set of holding conditions, both increases and decreases occurred in the diacetyl contents and also in the acetylmethylcarbinol contents; while some of the changes were too small to be of significance, others were relatively large. With each compound under each set of conditions the increases were more numerous than the decreases.

Because of the possibility of ripening butter to increase the diacetyl and acetylmethylcarbinol contents and improve the flavor, just as cream is ripened, the diacetyl and acetylmethylcarbinol contents of butter held 3 days at 60°F. plus 4 days at

36° to 40°F. and of butter held 1 week at 36° to 40° were compared. The results are given in the following summary:

Diacetyl	No. trials	Acetylmethylcarbinol	No. trials
Higher values with some holding at 60°F.			
Increases total	42	Increases total	40
0.10 p.p.m. or less	19	1.0 p.p.m. or less	8
.11 to .20 p.p.m.	10	1.1 to 3.0 p.p.m.	12
.21 to .30 p.p.m.	8	3.1 to 18.2 p.p.m.	20
.31 to .69 p.p.m.	5		
Same value with some holding at 60°F.			
Total	1	Total	4
Lower values with some holding at 60°F.			
Decreases total	13	Decreases total	11
0.10 p.p.m. or less	7	1.0 p.p.m. or less	0
.11 to .20 p.p.m.	3	1.1 to 3.0 p.p.m.	4
.21 to .30 p.p.m.	0	3.1 to 12.8 p.p.m.	7
.31 to .64 p.p.m.	3		

The summary shows that the short holding at 60°F. resulted in a higher diacetyl content in 42 (75%) of the 56 trials, no change in one trial (2%) and a decrease in 13 trials (23%). The acetylmethylcarbinol content was higher in 40 (73%) of the 55 trials, the same in 4 trials (7%) and lower in 11 trials (20%).

DISCUSSION

Since production of diacetyl and acetylmethylcarbinol in ripening cream is brought about by action of the butter culture organisms and is influenced by a number of factors, some variation in the results of comparable trials would be expected, just as with many other biological processes. Differences in the activities of the various butter cultures and in the citric acid contents of the cream, especially sour cream where some of the citric acid probably is destroyed during the souring, may have been factors in the irregularities.

The general effects of an increase in the cream acidity, addition of citric acid to the cream and agitation of the cream during ripening in producing higher diacetyl and acetylmethylcarbinol contents in ripening cream are in agreement with the results of various studies on butter cultures. Michaelian, Farmer and Hammer (6) noted that in a butter culture most of the acetylmethylcarbinol plus diacetyl was produced relatively late in the ripening. They also reported that butter culture organisms could destroy acetylmethylcarbinol plus diacetyl, with high acidities tending to retard the destruction and neutralization tending to accelerate it. Hammer (3) indicated the importance of citric acid as a source of volatile acids in butter cultures, and the significance of this compound from the standpoint of development of flavoring materials in butter cultures has been emphasized by other investigators. In butter cultures that were aerated by shaking, Prill and Hammer (11) obtained increases in diacetyl on holding. Virtanen (15) observed that if the ripening process was conducted in thin layers, the production of diacetyl and acetylmethylcarbinol was especially active. A large increase in production of diacetyl and acetylmethylcarbinol in butter cultures by aeration under pressure was reported by Brewer *et al.* (1).

Variations in the results obtained with different butter cultures are in agreement with commercial experience; also, they emphasize the necessity of proper selection of the cultures to be used in manufacture of unsalted butter.

The general effects of certain cream-ripening procedures in increasing the diacetyl and acetylmethylcarbinol contents of cream and also the score of butter held under certain conditions indicate the importance of diacetyl as a flavor constituent of butter. Irregularities between diacetyl contents of the cream and scores of the butter involve the degree of retention of diacetyl in the butter and also overshadowing of the desirable effect of diacetyl through off-flavors of various types in the butter; presumably the off-flavors involve both bacteriological and chemical changes.

Variations in the ratios of the diacetyl and acetylmethylcarbinol contents of the cream and of the corresponding butter suggest that significant differences occur in the churning process, even when an attempt is made to carry it out on a uniform basis. Differences probably include not only the degree of retention of buttermilk in the butter but the rates of diacetyl and acetylmethylcarbinol production during the churning process (12) and the loss of these materials during the washing of butter (8). Hoecker and Hammer (4) also reported variations in the degree of retention of diacetyl and acetylmethylcarbinol during churning.

Increases and decreases in the diacetyl and acetylmethylcarbinol contents of butter under various holding conditions emphasize that changes in these compounds go on in butter just as they do in a butter culture. The increases presumably play a part in the flavor development that unsalted butter made with butter culture so commonly shows, especially for a short period immediately after it is made, while the decreases may explain the rapid loss of flavor that this type of butter sometimes undergoes. It may be that with unsalted butter a short ripening period on a rather standardized basis for the butter itself would be advantageous from the standpoint of flavor development (14).

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TABLE 1. DIACETYL (Ac_2) AND ACETYLMETHYLCARBINOL (Amc) CONTENTS OF SWEET CREAM RIPENED TO VARIOUS ACIDITIES.

Laboratory trials											
	Cream						Cream				
Trial no.	Ripening temp.	% acid	pH	Ac_2 p.p.m.	Amc p.p.m.	Trial no.	Ripening temp.	% acid	pH	Ac_2 p.p.m.	Amc p.p.m.
1	70°F.	0.37	5.13	0.84	35.6	8	70°F.	0.35	5.54	0.52	33.8
	70	.44	4.89	1.49	42.9		70	.40	5.32	.57	38.3
	70	.51	4.67	2.28	52.4		70	.44	5.09	.72	45.4
2	70	.33	5.54	.46	30.9	9	65	.42	5.02	.90	36.8
	70	.38	5.43	.59	34.1		65	.47	4.81	1.05	39.4
	70	.44	5.14	.74	40.7		65	.51	4.77	1.05	42.4
							65	.56	4.73	1.75	55.4
3	70	.35	5.60	.27	24.8	10	65	.30	5.47	.71	19.4
	70	.38	5.50	.34	25.9		65	.36	5.23	.74	17.3
	70	.42	5.45	.39	27.2		65	.40	5.15	.72	12.0
							65	.44	5.00	.75	12.0
4	70	.34	5.71	.52	39.6	11	65	.46	4.77	1.14	60.0
	70	.40	5.45	.60	41.3		65	.50	4.75	1.30	61.2
	70	.44	5.35	.63	41.7		65	.53	4.71	2.78	68.6
							65	.59	4.67	2.32	85.8
5	70	.33	5.63	.48	37.6	12	60	.34	5.61	.40	23.7
	70	.39	5.49	.50	38.5		60	.38	5.55	.47	25.9
	70	.44	5.34	.61	42.2		60	.42	5.50	.32	27.3
6	70	.35	5.24	.75	34.8	13	60	.37	5.57	.57	26.8
	70	.42	5.18	1.05	37.7		60	.49	5.12	.56	29.8
	70	.49	4.48	1.67	50.4		60	.54	4.98	.44	29.0
7	70	.41	5.40	.31	25.9	14	60	.33	5.78	.58	43.8
	70	.50	5.12	.37	28.0		60	.39	5.45	.59	44.9
	70	.55	4.95	.44	29.0		60	.45	5.28	.60	47.3

15	60	.38	5.22	1.40	34.9
	60	.44	5.05	1.45	38.1
	60	.49	4.84	1.70	52.4
16	60	.38	5.12	1.02	36.3
	60	.46	4.89	1.52	41.4
	60	.52	4.70	1.77	52.5
17	60	.35	5.48	.62	28.0
	60	.41	5.29	.84	28.5
	60	.46	5.01	.98	32.6
18	60	.36	5.42	.84	44.4
	60	.40	5.25	.85	44.6
	60	.45	5.09	.97	48.4
19	60	.32	5.78	.66	44.4
	60	.39	5.52	.70	45.4
	60	.44	5.38	.72	52.1
20	32	.14	6.46	.28	16.5
	55	.28	5.49	.56	19.9
	55	.34	5.34	.82	13.1
	55	.39	5.21	.86	15.2
	55	.44	5.09	.60	15.5
21	32	.19	5.62	.27	10.0
	55	.41	5.11	.87	31.4
	55	.44	5.05	.97	31.4
	55	.51	4.86	1.03	39.5
	55	.54	4.80	1.17	40.8
22	55	.44	4.91	1.44	59.1
	55	.49	4.79	1.54	60.6
	55	.53	4.77	1.73	65.7
	55	.58	4.73	2.06	74.1

23	55	.27	5.65	.59	21.6
	55	.35	5.33	.69	24.9
	55	.40	5.03	.72	26.9
	55	.44	4.82	.84	34.0
24	32	.18	6.43	.20	9.9
	55	.29	5.53	.62	29.2
	55	.35	5.38	.71	31.2
	55	.43	5.21	.75	31.2
	55	.48	4.92	.84	32.4
25	32	.10	6.54	.23	3.5
	55	.36	5.06	.90	34.5
	55	.42	4.97	1.11	37.7
	55	.48	4.84	1.20	46.7
	55	.52	4.78	1.84	52.3
26	32	.20	6.39	.21	9.4
	55	.41	5.15	1.13	39.9
	55	.50	4.85	1.72	51.3
	55	.55	4.75	2.02	69.6
	55	.60	4.69	3.86	81.0
27	32	.18	6.57	.12	14.1
	55	.30	5.85	.46	2.5
	55	.34	5.81	.27	2.5
	55	.42	5.67	.24	2.3
	55	.45	5.56	.19	2.2
28	55	.28	5.80	.59	25.9
	55	.33	5.61	.65	25.9
	55	.36	5.42	.72	30.6
	55	.43	5.19	.74	35.0
29	55	.35	5.17	.57	21.9
	55	.45	5.05	.67	22.4
	55	.51	4.87	.56	23.9
	55	.56	4.74	1.12	35.8
30	55	.35	5.72	.56	45.4
	55	.39	5.56	.59	46.0
	55	.43	5.35	.60	47.0
	55	.45	5.36	.61	50.4

TABLE 1. DIACETYL (Ac_2) AND ACETYLMETHYLCARBINOL (Amc) CONTENTS OF SWEET CREAM RIPENED TO VARIOUS ACIDITIES—Continued.

Laboratory trials											
	Cream						Cream				
Trial no.	Ripening temp.	% acid	pH	Ac_2 p.p.m.	Amc p.p.m.	Trial no.	Ripening temp.	% acid	pH	Ac_2 p.p.m.	Amc p.p.m.
31	50°F.	0.33	5.62	0.89	42.9	34	50°F.	0.37	5.13	1.12	35.6
	50	.39	5.42	.90	44.0		50	.45	4.99	1.14	38.6
	50	.46	5.17	1.12	45.7		50	.50	4.85	1.85	58.8
32	50	.40	5.36	.60	27.4	35	50	.33	5.68	.55	25.8
	50	.51	5.04	.82	32.2		50	.39	5.52	.62	27.9
	50	.58	4.90	1.04	35.2		50	.43	5.43	.64	30.5
33	50	.33	5.66	.79	40.2	36	50	.40	4.96	1.32	37.2
	50	.40	5.40	.85	42.7		50	.44	4.84	1.63	46.6
	50	.48	5.20	1.00	44.8		50	.50	4.72	2.03	53.7

TABLE 2. DIACETYL (Ac_2) AND ACETYLMETHYLCARBINOL (Amc) CONTENTS OF NEUTRALIZED SOUR CREAM RIPENED TO VARIOUS ACIDITIES.

Laboratory trials					
	Cream				
Trial no.	Ripening temp.	% acid	pH	Ac_2 p.p.m.	Amc p.p.m.
1	32° F.	0.15	6.89	0.37	22.2
	62	.34	5.23	1.35	27.2
	62	.36	5.10	1.85	39.8
	62	.39	5.00	2.11	55.6
	62	.42	4.95	2.57	75.3
2	32	.14	6.75	.35	23.3
	60	.32	5.70	1.41	43.1
	60	.37	5.14	1.81	44.2
	60	.42	4.85	1.90	50.6
3	32	.14	6.69	.55	20.9
	58	.31	5.36	1.40	23.8
	58	.40	4.99	1.73	24.0
	58	.44	4.79	2.11	24.3

TABLE 3. DIACETYL (Ac_2) AND ACETYLMETHLYCARBINOL (Amc) CONTENTS OF SWEET (Swt.) OR NEUTRALIZED SOUR (Sr.) CREAM RIPENED TO VARIOUS ACIDITIES.

Semi-commercial trials

Trial no.	Cream					
	Type of cream	Ripening temp.	% acid	pH	Ac_2 p. p. m.	Amc p. p. m.
1	Swt.	53°F.	0.36	5.39	0.44	11.8
	Swt.	53	.42	5.23	.48	11.9
	Swt.	53	.49	4.91	.54	15.3
2	Swt.	53	.35	5.34	.43	17.9
	Swt.	53	.40	5.26	.60	24.0
	Swt.	53	.45	5.17	.72	38.5
3	Swt.	53	.35	5.33	.46	20.4
	Swt.	53	.42	5.12	.68	25.7
	Swt.	53	.48	4.95	.97	30.1
4	Swt.	50	.48	4.79	4.23	37.4
	Swt.	50	.52	4.73	4.80	101.4
5	Swt.	52	.48	4.90	2.94	87.4
	Swt.	52	.51	4.83	3.78	62.2
6	Swt.	54	.35	5.33	1.19	28.9
	Swt.	54	.40	5.15	1.23	32.5
7	Swt.	55	.38	4.98	1.24	23.8
	Swt.	55	.46	4.85	2.15	32.8
8	Sr.	51	.31	5.35	1.55	62.0
	Sr.	51	.35	5.26	1.95	72.4
9	Sr.	after past.	.23	6.52	.21	21.7
	Sr.	plus culture	.26	6.47	.28	34.0
	Sr.	54	.32	5.92	.67	34.1
	Sr.	54	.38	5.69	.76	31.7
10	Sr.	after past.	.17	6.58	.56	29.8
	Sr.	plus culture	.18	6.52	.62	36.0
	Sr.	53	.37	5.29	1.88	64.7
	Sr.	53	.49	4.98	1.49	28.8

TABLE 4. DIACETYL (Ac_2) AND ACETYLMETHYLCARBINOL (Amc) CONTENTS OF SWEET CREAM RIPENED WITH AND WITHOUT ADDED CITRIC ACID TO ABOUT THE SAME ACIDITIES.

Laboratory trials

	Cream					
Trial no.	% citric acid added	Ripen-ing temp.	% acid	pH	Ac_2 p. p. m.	Amc p. p. m.
1	0.00	55°F.	0.36	5.06	0.90	34.5
	.05	55	.35	5.15	1.01	32.6
2	.00	55	.42	4.97	1.11	37.7
	.05	55	.40	5.02	1.45	36.5
3	.00	55	.48	4.85	1.20	46.7
	.05	55	.46	4.91	1.49	49.2
4	.00	55	.52	4.78	1.84	52.3
	.05	55	.50	4.81	2.52	55.2
5	.00	55	.30	5.85	.47	12.5
	.05	55	.32	5.73	.98	18.5
6	.00	55	.34	5.81	.27	2.5
	.05	55	.35	5.64	.98	13.1
7	.00	55	.36	5.67	.24	2.4
	.05	55	.36	5.61	.63	5.2
8	.00	55	.37	5.56	.18	2.2
	.05	55	.37	5.51	.56	2.7
9	.00	55	.56	4.74	1.19	35.8
	.05	55	.57	4.72	1.27	44.4
10	.00	55	.39	5.19	.75	35.0
	.05	55	.39	5.19	.78	58.6
11	.00	54	.30	5.69	.20	10.2
	.05	54	.31	5.65	.25	12.1
12	.00	54	.34	5.52	.19	10.8
	.05	54	.34	5.55	.20	12.5
13	.00	55	.35	5.56	.59	46.0
	.05	55	.34	5.58	.66	54.4
14	.00	60	.42	5.02	.87	37.9
	.10	60	.41	5.01	1.09	45.1

TABLE 5. DIACETYL (Ac_2) AND ACETYLMETHYLCARBINOL (Amc) CONTENTS OF NEUTRALIZED SOUR CREAM RIPENED WITH VARIOUS AMOUNTS OF ADDED CITRIC ACID TO ABOUT THE SAME ACIDITIES.

Laboratory trials

Trial no.	Cream					
	% citric acid added	Ripen-ing temp.	% acid	pH	Ac_2 p. p. m.	Amc p. p. m.
1	0.00	60°F.	0.37	5.14	1.80	44.2
	.05	60	.36	5.14	1.80	48.0
	.10	60	.38	5.09	1.80	57.2
	.15	60	.39	4.99	2.02	58.3
2	.00	60	.44	4.75	1.81	37.4
	.05	60	.45	4.76	2.44	56.2
	.10	60	.47	4.68	2.72	72.2
	.15	60	.44	4.74	2.72	78.0
3	.00	58	.40	4.99	1.73	24.3
	.05	58	.37	5.20	1.57	32.3
	.10	58	.39	5.05	2.00	44.4
	.15	58	.42	4.93	2.17	48.6
4	.00	58	.44	4.79	1.70	24.0
	.05	58	.45	4.76	2.62	44.9
	.10	58	.45	4.75	3.09	59.3
	.15	58	.45	4.74	3.24	64.9
5	.00	63	.43	4.73	1.59	33.2
	.10	63	.44	4.71	2.66	73.2
6	.00	63	.47	4.67	2.90	33.9
	.10	63	.49	4.63	3.76	77.4
7	.00	62	.42	4.92	1.41	31.1
	.05	62	.43	4.92	2.57	38.1
	.10	62	.45	4.90	3.37	76.8
	.15	62	.48	4.82	3.93	80.4
8	.00	62	.42	4.92	1.41	31.1
	.05	62	.37	5.10	1.85	39.8
	.10	62	.39	5.00	2.11	55.6
	.15	62	.42	4.95	2.57	75.3

TABLE 6. DIACETYL (Ac_2) AND ACETYLMETHYLCARBINOL (Amc) CONTENTS OF SWEET (Swt.) OR NEUTRALIZED SOUR (Sr.) CREAM RIPENED WITH AND WITHOUT ADDED CITRIC ACID TO ABOUT THE SAME ACIDITIES.

Semi-commercial trials							
	Cream						
Trial no.	Type of cream	% citric acid added	Ripening temp.	% acid	pH	Ac_2 p. p. m.	Amc p. p. m.
1	Swt.	0.00	52°F.	0.48	4.76	3.28	76.1
	Swt.	.05	52	.51	4.70	4.44	108.7
2	Swt.	.00	50	.48	4.79	4.23	37.4
	Swt.	.05	50	.53	4.71	4.10	21.6
3	Swt.	.00	53	.42	5.29	1.50	46.2
	Swt.	.05	53	.44	5.24	2.28	59.0
4	Swt.	.00	51	.51	4.83	3.78	62.2
	Swt.	.05	51	.53	4.77	5.90	97.0
5	Sr.	.00	51	.31	5.35	1.55	62.0
	Sr.	.05	51	.34	5.20	1.67	62.0
6	Sr.	.00	54	.32	5.92	.67	34.0
	Sr.	.05	54	.35	5.55	.82	30.0
7	Sr.	.00	53	.37	5.34	1.40	39.3
	Sr.	.05	53	.37	5.31	1.60	33.2

TABLE 7. DIACETYL (Ac_2) AND ACETYLMETHYLCARBINOL (Amc) CONTENTS OF SWEET (Swt.) OR NEUTRALIZED SOUR (Sr.) CREAM RIPENED WITH AND WITHOUT AGITATION.

Laboratory trials							
	Cream						
Trial no.	Type of cream	Agitation	Ripening temp.	% acid	pH	Ac_2 p. p. m.	Amc p. p. m.
1	Swt.	—	60°F.	0.25	6.31	0.53	42.7
	Swt.	+	60	.26	6.13	.84	106.6
2	Swt.	—	60	.52	4.81	2.35	72.7
	Swt.	+	60	.52	4.80	4.44	110.0
3	Swt.	—	60	.55	4.67	1.40	54.0
	Swt.	+	60	.56	4.69	3.35	108.0
4	Swt.	—	70	.26	6.01	.43	28.9
	Swt.	+	70	.26	6.01	1.22	59.9
5	Swt.	—	70	.35	5.65	.52	31.3
	Swt.	+	70	.35	5.68	1.35	78.3
6	Swt.	—	60	.27	5.32	.78	30.6
	Swt.	+	60	.26	5.46	1.08	42.6
7	Swt.	—	60	.47	5.02	.87	37.9
	Swt.	+	60	.45	5.20	1.61	56.3
8	Sr.	—	63	.43	4.73	1.59	33.2
	Sr.	+	63	.44	4.78	2.40	46.1
9	Sr.	—	63	.47	4.67	2.90	33.9
	Sr.	+	63	.46	4.70	3.26	51.8

TABLE 8. DIACETYL (Ac_2) AND ACETYLMETHYLCARBINOL (Amc) CONTENTS OF SWEET (Swt.) OR NEUTRALIZED SOUR (Sr.) CREAM RIPENED WITH AND WITHOUT AGITATION AFTER ADDITION OF 0.1 PERCENT CITRIC ACID.

Laboratory trials

	Cream						
Trial no.	Type of cream	Agitation	Ripening temp.	% acid	pH	Ac_2 p. p. m.	Amc p. p. m.
1	Swt. Swt.	— +	70°F. 70	0.38 .37	5.34 5.49	0.77 1.46	39.6 79.5
2	Swt. Swt.	— +	70 70	.45 .47	4.98 5.14	.91 1.90	42.7 96.9
3	Swt. Swt.	— +	60 60	.41 .40	5.01 5.02	1.09 1.57	45.1 61.0
4	Swt. Swt.	— +	60 60	.49 .46	4.74 4.92	1.73 2.75	65.3 101.2
5	Sr. Sr.	— +	63 63	.47 .46	4.71 4.71	2.66 3.50	73.2 83.9
6	Sr. Sr.	— +	63 63	.50 .50	4.63 4.65	3.27 5.60	77.4 108.7

TABLE 9. DIACETYL (Ac_2) AND ACETYLMETHYLCARBINOL (Amc) CONTENTS OF SWEET (Swt.) OR NEUTRALIZED SOUR (Sr.) CREAM RIPENED WITH AND WITHOUT AGITATION.

Semi-commercial trials

	Cream						
Trial no.	Type of cream	Agitation	Ripening temp.	% acid	pH	Ac_2 p. p. m.	Amc p. p. m.
1	Swt. Swt.	— +	52°F. 52	0.48 .48	4.76 4.80	3.28 5.90	76.1 164.0
2	Swt. Swt.	— +	54 54	.35 .33	5.33 5.35	1.19 2.30	28.9 67.8
3	Swt. Swt.	— +	55 55	.38 .37	4.98 4.97	1.24 1.91	23.8 55.8
4	Sr. Sr.	— +	55 55	.37 .36	5.34 5.41	1.40 2.12	39.3 78.0
5	Sr. Sr.	— +	53 53	.49 .45	4.98 5.15	1.50 1.63	28.8 39.1

TABLE 10. DIACETYL (Ac_2) AND ACETYLMETHYLCARBINOL (Amc) CONTENTS OF SWEET CREAM RIPENED AT DIFFERENT TEMPERATURES TO ABOUT THE SAME ACIDITIES.

	Laboratory trials										
	Cream						Cream				
Trial no.	Ripening temp.	% acid	pH	Ac_2 p. p. m.	Amc p. p. m.	Trial no.	Ripening temp.	% acid	pH	Ac_2 p.p.m.	Amc p.p.m.
1	50° F.	0.33	5.62	0.89	42.9	10	50° F.	0.33	5.68	0.55	25.8
	60	.32	5.78	.66	44.4		60	.34	5.61	.40	23.7
	70	.33	5.63	.48	37.5		70	.35	5.60	.28	24.8
2	50	.39	5.42	.89	45.0	11	50	.39	5.52	.62	27.9
	60	.39	5.52	.70	45.0		60	.38	5.55	.47	25.9
	70	.39	5.49	.49	38.5		70	.38	5.50	.34	25.9
3	50	.46	5.17	1.12	45.7	12	50	.43	5.43	.64	30.5
	60	.44	5.38	.72	52.1		60	.42	5.43	.39	27.3
	70	.44	5.34	.61	42.2		70	.42	5.45	.39	27.2
4	50	.33	5.66	.79	40.2	13	50	.40	4.96	1.32	37.3
	60	.33	5.78	.58	43.8		60	.38	5.12	1.02	36.3
	70	.34	5.71	.52	39.6		70	.37	5.13	.84	35.6
5	50	.40	5.40	.85	42.7	14	50	.44	4.84	1.63	46.6
	60	.39	5.45	.58	44.9		60	.46	4.89	1.52	41.4
	70	.40	5.45	.60	41.3		70	.44	4.89	1.49	43.0
6	50	.47	5.20	1.00	44.8	15	50	.51	4.72	2.03	53.7
	60	.45	5.28	.60	47.3		60	.52	4.70	1.77	52.5
	70	.44	5.35	.63	41.7		70	.51	4.67	2.28	52.4
7	50	.40	5.26	.60	27.4	16	50	.37	5.13	1.12	35.6
	60	.37	5.57	.57	26.8		60	.36	5.22	1.40	34.9
	70	.40	5.40	.31	26.0		70	.35	5.24	.75	34.8
8	50	.51	5.04	.82	32.2	17	50	.45	4.99	1.45	38.6
	60	.49	5.12	.56	29.8		60	.44	5.05	1.45	38.1
	70	.50	5.12	.37	28.0		70	.42	5.18	1.05	27.6
9	50	.58	4.90	1.04	35.2	18	50	.50	4.85	1.85	58.8
	60	.54	4.98	.44	29.0		60	.49	4.84	1.70	52.4
	70	.55	4.95	.44	29.0		70	.49	4.84	1.67	50.4

TABLE 11. DIACETYL (Ac_2) AND ACETYLMETHYLCARBINOL (Amc) CONTENTS OF SWEET (Swt.) OR NEUTRALIZED SOUR (Sr.) CREAM RIPENED AT DIFFERENT TEMPERATURES TO ABOUT THE SAME ACIDITIES.

Semi-commercial trials

	Cream					
Trial no.	Type of cream	Ripening temp.	% acid	pH	Ac_2 p. p. m.	Amc p. p. m.
1	Swt.	55°F.	0.55	4.95	2.57	36.8
	Swt.	62	.55	4.85	2.74	42.6
	Swt.	70	.57	4.72	2.99	45.8
2	Swt.	62	.55	4.65	1.95	42.9
	Swt.	70	.52	4.73	1.92	33.0
3	Swt.	61	.34	4.99	1.68	36.2
	Swt.	70	.36	4.95	.79	8.2
4	Sr.	62	.50	4.83	2.06	45.1
	Sr.	70	.51	4.75	2.13	46.2